BUSINESS CONFIDENTIAL PROJECT REPORT Copy II

CONTINUOUS PROCESS FOR THE PRODUCTION OF SILANE ESTERS

Distillation of Cyanoethyltrichlorosilane

AUTHOR:	C. J. Litteral	DATE:	April 25, 1968
PROJECT LEADER:	R. P. Boersma	PROJECT NUMBER:	336814
SUPERVISOR:	G. M. Omietanski	FILE NUMBER:	68-15

SUMMARY

Laboratory studies indicate that the available equipment in the Monomers Department is not suitable for distillation of cyanoethyltrichlorosilane. It is recommended that installation of suitable apparatus for high boiling chlorosilanes be given consideration. However, in the absence of such equipment it is recommended that existing or slightly modified monomers apparatus be used to remove lites, and that product distillation be accomplished, if possible, in suitable equipment in another area.

Research and Development Department
UNION CARBIDE CORPORATION
CHEMICALS AND PLASTICS
Sistersville, West Virginia

CONTINUOUS PROCESS FOR THE PRODUCTION OF SILANE ESTERS

INTRODUCTION

The Polymers Production Department has indicated that increased capacity and considerable annual savings could be realized in the cyanoethyltriethoxysilane (CNE) system if the starting cyanoethyltrichlorosilane (CNT) were free of lites and heavies.[1] Further, it has been suggested that a distilled material would be required should a continuous process unit be put into production.[2] A brief laboratory study was made to determine the suitability of using distillation equipment available in the monomers area for an overhead strip of lites and product.

DISCUSSION

An earlier attempt at CNT distillation in production equipment resulted in a gelled pot residue. However, laboratory distillation has been successful, when air was excluded, on a routine basis. A laboratory distillation of crude CNT, containing 10% Aroclor 1248, was lined out on the product fraction at 35 mm. Hg. 120°C bottom temperature, and held at reflux for 6 hours. The distillation was then completed, and shut down at a bottom temperature of 175°C. At this point, the residue which contained 5% CNT (3% of charged product) was a very low viscosity fluid. However, on cooling to room temperature, the residue viscosity increased to approximately 1-5 x 10^9 centipoises. These heavies can still be easily handled. Conditions in the production still (S-12) are expected to be much more severe than those experienced in the laboratory distillation. Two experiments (one containing powdered iron) were run in the laboratory to determine the behavior of the heavies under severe conditions. Product was distilled overhead and the heavies, containing Aroclor 1248 were refluxed at 200-210°C, 35 ± 5mm. Hg. Both systems gelled 2-1/2 hours after completion of the distillation. A sample of CNE heavies held at the same conditions remained fluid throughout a 6-hour test.

CONCLUSIONS AND RECOMMENDATIONS

From this study and past experience CNT heavies appear much more sensitive to heat than CNE heavies. An attempt to use a conventional reboiler overhead still, such as those available, is risky at best. However, it has been shown that an apparatus which will not require heavies to be held at high temperatures could be used reliably for CNT distillation. It is recommended that consideration be given to the installation of a wiped film or falling film unit for distillation of high boiling chlorosilanes. Until such an apparatus becomes available, a more suitable chlorosilane for CNE production could be prepared by lites stripping in available monomers apparatus or by modifying the reactor to allow removal of lites at the time of manufacture.

NOTEBOOK REFERENCE - Research and Development N.B. 1466

BIBLIOGRAPHY

1. Operations Improvement Proposal, M-45 - W. M. Stewart
2. Continuous Process for the Production of Silane Esters, C. J. Litteral, March 19, 1968.

C. J. Litteral

C. J. Litteral

CJL/lss